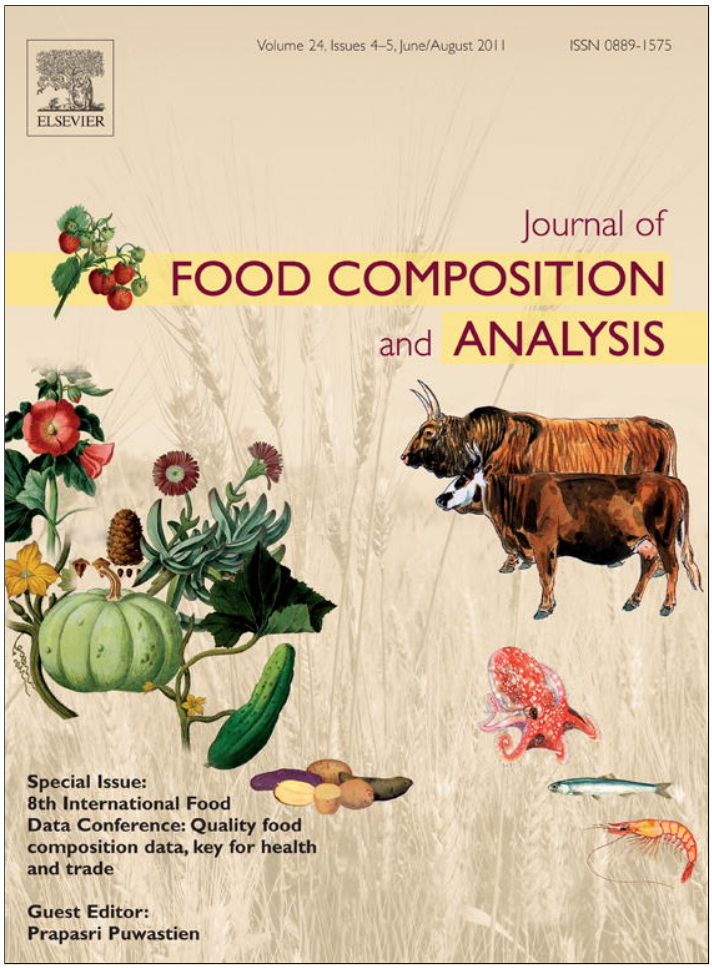

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Original Article

Method performance study for total solids and total fat in coconut milk and products

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ABSTRACT

Many analytical methods are available for total solids and total fat in various kinds of food. However, none is specific for coconut milk and products. The aim of this study was to establish methods and to conduct the method performance study on determination of total solids and total fat in coconut milk and products. In the single laboratory study, three methods for total solids determination were conducted: drying in hot air oven at 102 ± 2 °C and at 130 ± 3 °C and drying in vacuum oven at 70 °C, 70 mmHg, as well as three methods for total fat determination: direct extraction method, acid hydrolysis method and alkaline hydrolysis method. Sixteen samples of aqueous coconut milk and coconut milk powder were selected as test materials. Total solids and total fat contents were analyzed in 10 individual samples of each test material ($N = 10$). The statistical analysis (one-way ANOVA) showed no significant difference for total solids and no difference for total fat content by the used methods ($p > 0.05$). Since the drying method by hot air oven at 102 ± 2 °C is the most commonly used for total solids in food laboratories, it was selected for further inter-laboratory study. For total fat determination, the alkaline hydrolysis method (Roese–Gottlieb) was selected for further inter-laboratory study since it is less time-consuming and involves fewer steps than the acid hydrolysis method. Seventeen laboratories participated in the inter-laboratory study and seven representative test materials with varying levels of total solids and fat were used. The method performance characteristics are presented. These methods were proposed to CODEX and endorsed as the CODEX standard methods (type I) for aqueous coconut milk and coconut cream analyses.

1. Introduction

In Thailand as well as other ASEAN (The Association of Southeast Asian Nations) countries, coconut milk is not only largely consumed, but also exported to other. For the latter reason, the Asian and Pacific Coconut Community (APCC) proposed standard for coconut milk and products to CODEX and in the year 2003 CODEX endorsed the standard as "CODEX STAN 240-2003: Codex standard for aqueous coconut milk and coconut cream products". According to this standard, aqueous coconut products are classified into 4 categories: light coconut milk, coconut milk, coconut cream and coconut cream concentrate depending on total solids and total fat content (CODEX STAN 240, 2003).

There are various methods for the determination of total solids and moisture content in foods according to AOAC International and ISO standards such as drying in hot air oven for fluid milk (AOAC, 2005; ISO 6731, 1989), cheese and processed cheese (AOAC, 2005; ISO 5534, 1985), flour (AOAC, 2005), cereal and cereal product (ISO 712, 2009), drying in vacuum oven for honey (AOAC, 2005), fruit and products (AOAC, 2005) and beverage (AOAC, 2005), distillation method for spices and condiments (AOAC, 2005; ISO 939, 1980) and chemical method or Karl Fisher Titration for water in dried vegetables (AOAC, 2005). A rapid moisture analyzer based on infrared and microwave drying (AOAC, 2005) is also accepted, mainly in-process, for quality control purpose (Bradley, 2003). For the determination of total fat, the solvent extraction methods are widely used such as direct solvent extraction technique for butter and spreadable fat (ISO 17189, 2003), acid hydrolysis–solvent extraction technique (Schmid–Bondzynski–Ratzlaff) for flour (AOAC, 2005), sea foods (AOAC, 2005) and alkaline hydrolysis–solvent extraction technique (Roese–Gottlieb) for milk (AOAC, 2005; ISO 1211, 1999) and cream (ISO 2450, 2008). Non-solvent extraction method for raw milk, Babcock method (AOAC, 2005), Gerber method (AOAC, 2005) and instrumental methods such as IR and NMR (Min and Boff, 2003) are also known such as for milk (AOAC, 2005). In spite of the existence of various methods for the matrices mentioned, and in spite of the CODEX standard for aqueous coconut milk and coconut cream products, there is no agreed method of analysis for total solids and for total fat in this matrix.

It is, therefore, necessary to have standard methods of analysis that can be agreed upon internationally, such as in cases of disputes on trade. In addition to single laboratory study, we report

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here a collaborative trial based on acceptable international protocols conducted to ensure the reproducibility of the methods when used by laboratories regardless of location or time. The method performances of the proposed methods were evaluated based on the guidelines for collaborative study procedures (Horwitz, 2005), and the results are reported. Thus two main objectives of this study were to find the appropriate methods for determination of total solids and total fat in coconut milk and products and to propose these methods for adoption by CODEX committee on methods of analysis and sampling (CCMAS).

2. Materials and methods

2.1. Single laboratory study

2.1.1. Test materials

Sixteen samples include 5 aqueous light coconut milks, 5 coconut milks, 5 coconut creams and 1 coconut milk powder were provided by four manufacturers in Thailand.

2.1.2. Selection of methods

Total solids. Three oven drying techniques with varying conditions were selected: (A) hot air oven 102 ± 2 °C, (B) hot air oven 130 ± 3 °C and (C) vacuum oven 70 °C, 70 mmHg.

Total fat. Three techniques of solvent extraction were selected: (A) direct extraction method: sample was extracted with petroleum ether, (B) acid hydrolysis–solvent extraction method (Schmid–Bondzynski–Ratzlaff principle): sample was digested with hydrochloric acid before extracted with ether and petroleum ether and (C) alkaline hydrolysis–solvent extraction method (Roese–Gottlieb principle): sample was digested with ammonium hydroxide before extracted with ether and petroleum ether.

2.1.3. Determination

Each sample was analyzed for total solids and total fat in 10 replicates.

2.1.4. Evaluation of the result

One-way ANOVA was used to evaluate the resulting data.

2.2. Inter-laboratory study:

The inter-laboratory study was conducted in 2 rounds. In the first round, three samples namely light coconut milk (1), coconut cream (1) and coconut milk powder (1) were analyzed in duplicate. Both results were reported. In the second round, the participants received 8 samples which were 4 pairs of blind duplicates. They were requested to report a single result for each sample. The collaborative study was conducted according to the guidelines for collaborative study procedures (Horwitz, 2005).

2.2.1. Collaborating laboratories

Seventeen laboratories in Thailand, 7 government and 10 private, were accepted to participate in the inter-laboratory study. All of them implement the quality system according to ISO 17025:2005 and have long-term experience in proximate analysis of food.

2.2.2. Preparation of test materials

Round I. There were three samples which included 1 light coconut milk, 1 coconut cream and 1 coconut milk powder.

Round II. There were eight samples (four pairs of blind duplicates) which included light coconut milk, coconut milk, coconut cream and coconut milk powder. Each sample (ca. 50 g) was filled into sealed plastic bottles. The summary of the sampling procedure used in the collaborative study is shown in Table 1.

2.2.3. Homogeneity testing

Ten bottles from each of the prepared samples were selected at random. The total solids and total fat in each selected bottle were determined in duplicate, and the data were evaluated by one-way ANOVA according to the international harmonized protocol (Thompson et al., 2006).

2.2.4. Distribution of samples and documents

One bottle of each sample accompanied by six documents of instruction for participants, sample receipt form, report form of final results, questionnaires for the methods and 2 determination procedures were sent to laboratories by hand. Each laboratory was assigned a laboratory code number which was kept confidential.

In the instruction sheets, the participants were asked to store the test materials at 2–8 °C immediately after receiving and during the process, to analyze the test material within 2 days and report the results in the report form with unit of expression and number of significant decimal places as indicated. They were advised to follow the procedures exactly as defined.

2.2.5. Submission of the results

The participants were asked to submit the results and related documents within 3 weeks.

2.2.6. Analytical method

Total solids determination. One to five grams of sample (approximately 5 g for light coconut milk, 3 g for coconut milk, 2 g for coconut cream and 1 g for coconut milk powder) was accurately weighed into a pre-weighed round flat-bottomed metal dish provided with a fitting lid (about 5 cm diameter). The uncovered

Table 1
Summary of the sampling procedure used in the collaborative study.

Round no.	Sample code	Sample type	Sample description	Total solids content (%)	Fat content (%)
Round I	A	Single sample	Light coconut milk	<13	<10
	B	Single sample	Coconut cream	26–37	20–28
	C	Single sample	Coconut milk powder	>80	>30
Round II	No. 1 No. 5	Blind duplicates	Light coconut milk	<13	<10
	No. 2 No. 6	Blind duplicates	Coconut milk	13–25	10–19
	No. 3 No. 7	Blind duplicates	Coconut cream	26–37	20–28
	No. 4 No. 8	Blind duplicates	Coconut milk powder	>80	>30

dish was placed on a boiling-water bath for 30 min or until most of the moisture was driven off. The bottom of the dish was then wiped off and the dish was transferred to a well-ventilated oven at 102 ± 2 °C. The lid was placed next to the dish in the oven. They were dried for 2 h in the oven and then the dish was covered with the lid, cooled for 30 min in a desiccator and weighed. The dish and the lid was heated again for 30 min periods in the oven, cooled and weighed until the difference between the two successive weightings did not exceed 1 mg.

Total fat determination. One to six grams of sample (approximately 6 g for light coconut milk, 3 g for coconut milk, 2 g for coconut cream and 1 g for coconut milk powder) was accurately weighed into the fat extraction flask. Water was added to complete the volume to 10 ml and mixed. Then 1.5 ml of ammonium hydroxide was added and mixed followed by 10 ml of alcohol (95%) and the contents were again well mixed. Twenty five milliliters of peroxide-free diethyl ether was added to the flask, closed with the stopper then it was shook vigorously for 1 min. Twenty five milliliters of light petroleum ether (b.p. 40–60 °C) was added and the flask was shook vigorously for 1 min. After separation was complete (after standing or by centrifuging at 600 rpm for 30 s) the fat solution was transferred into a suitable dish (previously dried in hot air oven for 1 h at 102 ± 2 °C, cooled to room temperature in a desiccator and weighed). To the flask, 5 ml ethanol was added and the contents mixed. The extraction with 15 ml of ether and with 15 ml of light petroleum ether followed by the subsequent operations was repeated. The final extraction was done with the ethers but without alcohol. The solvents from the dish were evaporated and the dish dried to constant weight in hot air oven at 102 ± 2 °C for 1 h, cooled to room temperature in a desiccator (ca. 30 min) and weighed. If any non-fatty matter appeared to be present, then the fat would be washed out from the flask with light petroleum, dried, reweighed and the result was corrected accordingly. A pair of reagent blanks was included and the residue should be less than 0.0020 g.

2.2.7. Statistical analysis of results

Outliers test. The results were examined for evidence of individual systematic error using Cochran's and Grubbs' test, using procedures described in the guidelines for collaborative study procedures (Horwitz, 2005).

Repeatability and reproducibility. Calculations for repeatability (r) and reproducibility (R) as well as repeatability relative standard deviation (RSD_r), reproducibility relative standard deviation (RSD_R), repeatability standard deviation (S_r) and reproducibility standard deviation (S_R) were carried out on data remaining after removal of outliers.

3. Results and discussion

3.1. Single laboratory study

3.1.1. Total solids determination

The total solids levels in the 16 test materials are reported in Table 2. By using analysis of variance, the levels of total solids derived from the three methods of analysis were not significantly different ($p > 0.05$). However, since hot air oven is a basic instrument for all laboratories, it is cheaper and easier to operate and calibrate than a vacuum oven, and as drying at 102 °C is the general principle for moisture determination in foods, the method of drying in hot air oven at 102 °C was chosen as the candidate method for further collaborative study.

3.1.2. Total fat determination

The results for total fat are summarized in Table 3. Although the levels of total fat derived from the three methods of analysis were not significantly different ($p > 0.05$), the levels derived from direct solvent extraction method in 16 test materials were lower than those derived from acid hydrolysis and alkaline hydrolysis method. Between the alkaline hydrolysis–solvent extraction method and the acid hydrolysis–solvent extraction method, it is obvious that the former is easier to perform and involves fewer steps than the latter. Therefore, the alkaline hydrolysis–solvent extraction method was chosen as a candidate method for further collaborative study.

3.2. Inter-laboratory study

3.2.1. Homogeneity test

The result of the one way analysis of variance showed that all test values (F) were smaller than the critical F-value ($\alpha = 0.05$), implying no statistically significant difference between the bottles. This confirmed that the prepared materials were sufficiently homogeneous.

3.2.2. Outliers test

Collaborative study results for each material are presented in Tables 4–7. Data analysis was conducted using the guidelines for collaborative study procedures (Horwitz, 2005).

Table 2
Single laboratory analysis: summary for total solids levels in the 16 test materials.

No.	Total solids (g/100 g), mean ± SD, $N = 10$		
	Hot air oven 102 ± 2 °C	Hot air oven 130 ± 3 °C	Vacuum oven 70 °C, 70 mmHg
1	7.68 ± 0.01	7.63 ± 0.01	8.38 ± 0.02
2	8.66 ± 0.01	8.66 ± 0.02	8.67 ± 0.14
3	9.05 ± 0.02	9.05 ± 0.02	9.10 ± 0.02
4	7.82 ± 0.02	7.84 ± 0.01	7.84 ± 0.01
5	12.14 ± 0.02	12.06 ± 0.02	12.19 ± 0.02
6	18.86 ± 0.02	18.59 ± 0.02	18.77 ± 0.02
7	20.47 ± 0.03	20.35 ± 0.02	20.72 ± 0.21
8	21.87 ± 0.02	21.66 ± 0.02	21.69 ± 0.02
9	22.17 ± 0.05	22.05 ± 0.01	22.34 ± 0.03
10	24.69 ± 0.01	24.64 ± 0.01	24.72 ± 0.02
11	25.35 ± 0.02	25.24 ± 0.02	25.42 ± 0.02
12	26.32 ± 0.02	26.32 ± 0.02	26.46 ± 0.01
13	26.43 ± 0.02	26.85 ± 0.03	26.66 ± 0.03
14	29.24 ± 0.01	28.13 ± 0.02	27.52 ± 0.01
15	27.96 ± 0.03	27.80 ± 0.02	28.05 ± 0.02
16	98.92 ± 0.02	98.93 ± 0.03	98.95 ± 0.03

Table 3
Single laboratory analysis: summary for total fat levels in the 16 test materials.

No	Fat (g/100 g), mean ± SD, $N = 10$		
	Direct extraction	Acid hydrolysis	Alkaline hydrolysis
1	4.65 ± 0.02	4.97 ± 0.01	5.07 ± 0.02
2	5.01 ± 0.02	5.10 ± 0.02	5.06 ± 0.01
3	6.03 ± 0.02	6.37 ± 0.04	6.38 ± 0.12
4	6.22 ± 0.03	6.37 ± 0.04	6.40 ± 0.10
5	7.43 ± 0.02	8.92 ± 0.02	9.19 ± 0.01
6	14.55 ± 0.01	14.76 ± 0.01	15.05 ± 0.11
7	15.72 ± 0.01	16.97 ± 0.02	17.50 ± 0.02
8	17.52 ± 0.01	17.67 ± 0.02	17.60 ± 0.01
9	19.20 ± 0.02	19.34 ± 0.02	20.00 ± 0.10
10	19.72 ± 0.02	21.35 ± 0.01	21.80 ± 0.02
11	20.36 ± 0.02	21.02 ± 0.02	21.00 ± 0.27
12	20.45 ± 0.02	20.64 ± 0.01	20.53 ± 0.03
13	21.47 ± 0.01	22.88 ± 0.01	22.27 ± 0.03
14	21.49 ± 0.02	21.76 ± 0.01	21.76 ± 0.16
15	22.17 ± 0.02	22.52 ± 0.01	21.74 ± 0.02
16	41.44 ± 0.03	41.52 ± 0.02	41.64 ± 0.03

Table 4
Inter-laboratory study: total solids contents, g/100 g, obtained from drying at 102 ± 2 °C (round I).

Lab no.	Light coconut milk		Coconut cream		Coconut milk powder	
	A_1	A_2	B_1	B_2	C_1	C_2
1	8.75	8.73	27.94	27.97	99.09	99.02
2	8.89	8.91	27.31	27.20	98.70	98.75
3	8.87	8.82	27.81	26.95	98.89	98.87
4	8.57(G)	8.59(G)	25.94	26.53	98.71	98.70
5	8.79	8.77	26.12	27.29	98.88	98.80
6	8.86	8.86	27.01	27.09	98.67	98.68
7	8.79	8.83	26.63	26.14	98.77	98.82
8	8.79	8.79	26.96	27.07	98.58	98.56
9	8.86	8.86	25.48	27.33	98.79	98.78
10	8.88	8.84	27.09	27.10	98.49	98.55
11	8.88	8.90	27.31	27.35	98.98	99.13
12	8.84	8.85	27.08	26.95	98.74	98.79
13	8.82	8.84	26.87	26.87	98.73	98.69
14	8.85	8.86	27.31	28.71	98.75	98.72
15	8.90	8.87	26.39	26.78	99.32	99.42
16	8.82	8.81	26.90	26.79	99.02	99.06
17	8.82	8.86	26.96	27.09	98.68	98.75

(G) = outlier-between lab variation, evaluated by Grubbs test (2-tail; $\alpha = 0.025$).

Table 5
Inter-laboratory study: total solids contents, g/100 g, obtained from drying at 102 ± 2 °C (round II).

Lab no.	Light coconut milk		Coconut milk		Coconut cream		Coconut milk powder	
	No. 1	No. 5	No. 2	No. 6	No. 3	No. 7	No. 4	No. 8
1	10.56	10.57	20.66	20.70	28.58	28.80	99.13	99.13
2	10.60	10.62	21.04	20.73	29.12	29.06	99.04	98.93
3	10.32	10.41	20.38(G)	19.52(G)	28.31	28.43	99.35	99.31
4	9.92(C)	10.51(C)	20.71	20.59	28.99	28.98	98.98	98.74
5	10.49	10.46	20.98	20.95	36.27(C)	28.89(C)	99.22	99.25
6	10.51	10.56	20.86	20.74	29.88(C)	28.60(C)	98.62	98.94
7	10.62	10.61	20.89	20.99	28.89	29.11	99.17	99.09
8	10.64	10.64	20.98	20.99	29.14	29.10	99.09	99.02
9	10.60	10.58	21.06	21.08	29.08	29.20	99.17	99.12
10	10.69	10.67	21.00	21.00	28.82	28.53	99.14	99.18
11	10.39	10.30	20.49	20.97	29.05	28.59	99.69	99.55
12	10.61	10.60	20.95	20.97	29.12	28.79	99.23	99.21
13	10.68	10.78	21.04(C)	20.29(C)	29.25	29.07	99.84	99.54
14	10.45	10.44	20.53(C)	21.54(C)	27.96	28.80	99.15	99.13
15	10.66	10.65	21.42	21.30	29.19	29.50	100.0	100.0
16	10.59	10.57	20.47	20.68	29.09	29.07	99.55	99.42
17	10.63	10.61	20.94	21.04	29.16	29.13	99.29	99.17

(C) = outlier-within lab variation using Cochran test (1-tail; $\alpha = 0.025$). (G) = outlier-between lab variation, evaluated by Grubbs test (2-tail; $\alpha = 0.025$).

Table 6
Inter-laboratory study: total fat contents, g/100 g, obtained from Roese–Gottlieb method (round I).

Lab no.	Light coconut milk		Coconut cream		Coconut milk powder	
	A_1	A_2	B_1	B_2	C_1	C_2
1	5.60	5.51	20.71	21.19	41.06(G)	41.05(G)
2	6.59(G)	6.48(G)	21.12	21.22	40.89(G)	40.76(G)
3	5.75	5.75	22.82	22.74	43.59(C)	42.81(C)
4	5.70	5.72	21.65	21.67	42.57	42.59
5	5.26(G)	5.33(G)	18.79(G)	17.76(G)	36.81(C)	37.94(C)
6	5.65	5.69	21.68	21.69	42.53	42.78
7	5.94	5.90	22.14	22.46	42.70	42.76
8	5.90	5.93	20.70	20.89	43.00	43.12
9	5.90	5.92	21.67	22.18	42.91	43.14
10	6.07	6.06	23.64	22.96	42.54	42.67
11	5.95	6.04	22.16	21.94	43.08	43.12
12	5.99	5.99	22.08	22.18	42.99	42.96
13	5.97	5.97	20.35	20.61	43.15	43.10
14	6.07	6.07	21.55	22.12	43.36	43.66
15	5.70	5.73	22.00	22.13	43.54	43.77
16	6.02	5.98	22.42	22.22	43.09	43.17
17	5.85	5.96	21.72	21.86	42.64	42.74

(C) = outlier-within lab variation using Cochran test (1-tail; $\alpha = 0.025$). (G) = outlier-between lab variation, evaluated by Grubbs test (2-tail; $\alpha = 0.025$).

Table 7
Inter-laboratory study: total fat contents, g/100 g, obtained from Roese–Gottlieb method (round II).

Lab no.	Light coconut milk		Coconut milk		Coconut cream		Coconut milk powder	
	No. 1	No. 5	No. 2	No. 6	No. 3	No. 7	No. 4	No. 8
1	6.42(C)	7.68(C)	15.96	15.81	21.51(G)	20.81(G)	39.09(G)	39.29(G)
2	4.14(G)	4.49(G)	12.37(C)	16.72(C)	21.57	22.43	30.07(G)	29.44(G)
3	8.04	8.64	16.67	19.40	22.55	23.08	41.83	42.47
4	8.24	8.26	16.99	17.17	22.77	23.16	42.24	42.24
5	8.35	8.35	17.06	17.22	-	23.35	42.87	42.96
6	8.17	8.18	15.79	14.80	22.78	22.87	35.25(C)	38.38(C)
7	8.28	8.28	16.91	14.05	22.32(C)	27.65(C)	42.68	42.75
8	8.27	8.28	17.61	17.41	23.24	23.39	42.21	42.09
9	8.24	8.27	17.19	17.14	23.04	23.17	42.04	42.36
10	8.13	8.02	16.69	16.94	22.65	22.59	41.53	41.60
11	8.24	8.17	16.66	17.13	23.59	22.84	42.09	42.14
12	8.30	8.26	17.06	17.07	23.23	23.20	42.48	42.48
13	8.38	8.37	17.59	16.96	23.62	23.59	42.81	42.84
14	7.70(G)	8.28(G)	16.49	17.15	22.62	23.11	42.17	42.06
15	7.81(G)	7.77(G)	16.96	17.18	20.87(G)	19.86(G)	43.62	43.10
16	8.71	8.21	17.46	16.81	23.62	23.50	42.74	42.86
17	8.30	8.32	16.97	16.91	23.09	23.14	42.47	42.53

(C) = outlier-within lab variation using Cochran test (1-tail; $\alpha = 0.025$). (G) = outlier-between lab variation, evaluated by Grubbs test (2-tail; $\alpha = 0.025$).

Table 8
Summary of outlier determinations in the total solids determination.

No. of laboratory	Light coconut milk		Coconut milk		Coconut cream		Coconut milk powder	
	Round I	Round II	Round I	Round II	Round I	Round II	Round I	Round II
Total	17	17	-	17	17	17	17	17
Cochran outliers	–	1	–	2	–	2	–	–
Grubbs outliers	1		–	1	–		–	–

Cochran, Grubbs and pair Grubbs outlier data were determined and removed from the set of sample data prior to further data analysis. The Cochran test flagged laboratories that showed significantly greater variability among within-laboratory replicate analysis than other laboratories. The Grubbs test flagged laboratories that showed significantly extreme averages among the average of between-laboratories replicate analysis than other laboratories.

Total solids. The results were evaluated statistically according to the guidelines for collaborative study procedures (Horwitz, 2005). The results of Cochran and Grubbs tests for outlier determination are included in Tables 4 (round I) and 5 (round II). In round I, there were no Cochran outlier detected and only one single Grubbs outlier was identified for light coconut milk from laboratory 4. In round II, there were 5 Cochran outliers as follow; 1 for light coconut milk from laboratory 4, 2 for coconut milk from laboratories 13 and 14, 2 for coconut cream from laboratories 5 and 6. There was only one single Grubbs outlier for coconut milk from laboratory 3. No outlier was observed for coconut milk powder. The summary of outlier determinations is shown in Table 8.

Total fat. In round I (Table 6) analysis of the collaborative study data found 7 results pairs that were identified as outliers by either the Cochran or Grubbs test. The Cochran test showed that total fat in coconut milk powder from laboratories 3 and 5 should be rejected. The single Grubbs test showed that total fat for coconut cream from laboratory 5 was to be rejected. The pair Grubbs test showed that total fat for light coconut milk from laboratories 2 and 5 and for coconut milk powder from laboratories 1 and 2 was to be rejected.

In round II (Table 7) data of coconut cream (materials 3 and 7) from laboratory 5 were removed prior to outlier test because they were incomplete results. Therefore, outlier tests were conducted on the data of 16 laboratories for coconut cream and on the data of 17 laboratories for other materials

There were 4 results pairs that were identified as outliers by Cochran test and 7 paired by Grubbs test as following; 1 Cochran from laboratories 1 and 3 Grubbs from laboratories 2, 14 and 15 for light coconut milk; 1 Cochran from laboratory 2 for coconut milk; 1 Cochran from laboratories 7 and 2 Grubbs from laboratories 1 and 15 for coconut cream; 1 Cochran from laboratories 6 and 2 Grubb from laboratories 1 and 2 for coconut milk powder. The summary of the outlier laboratories is shown in Table 9.

3.2.3. Repeatability and reproducibility

Mean value, repeatability standard deviation (S_r), repeatability relative standard deviation (RSD_r), repeatability limit (r), reproducibility standard deviation (S_R), reproducibility relative standard deviation, (RSD_R) and reproducibility limit (R) which were

Table 9
Summary of outlier determinations in the total fat determination.

No. of laboratory	Light coconut milk		Coconut milk		Coconut cream		Coconut milk powder	
	Round I	Round II	Round I	Round II	Round I	Round II	Round I	Round II
Total	17	17	–	17	17	16	17	17
Cochran outliers	–	1	–	1	–	1	2	1
Grubbs outliers	2	3	–	–	1	2	2	2

Table 10
Conclusion of method performance for total solids determination.

Test materials	Light coconut milk		Coconut milk		Coconut cream	Coconut milk powder	
Laboratories remaining after eliminating outliers	16	16	14	17	15	17	17
Mean value, %	8.84	10.57	20.90	27.01	28.93	98.82	99.25
Repeatability standard deviation, S_r, %	0.02	0.03	0.12	0.49	0.21	0.04	0.10
Repeatability relative standard deviation, RSD_r, %	0.21	0.30	0.60	1.83	0.74	0.04	0.10
Repeatability limit, r (=2.8S_r), %	0.05	0.09	0.35	1.38	0.6	0.12	0.28
Reproducibility standard deviation, S_R, %	0.04	0.11	0.22	0.60	0.33	0.21	0.31
Reproducibility relative standard deviation, RSD_R, %	0.50	1.05	1.06	2.24	1.12	0.21	0.32
Reproducibility limit, R (=2.8S_R), %	0.12	0.31	0.62	1.69	0.91	0.59	0.88

Table 11
Conclusion of method performance for total fat determination.

Test materials	Light coconut milk		Coconut milk		Coconut cream	Coconut milk powder	
Laboratories remaining after eliminating outliers	15	13	16	16	13	13	14
Mean value, %	5.88	8.28	16.85	21.83	23.02	42.90	42.44
Repeatability standard deviation, S_r, %	0.03	0.16	0.76	0.23	0.28	0.11	0.17
Repeatability relative standard deviation, RSD_r, %	0.59	1.88	4.49	1.04	1.22	0.26	0.41
Repeatability limit, r (=2.8S_r), %	0.10	0.44	2.12	0.63	0.79	0.31	0.49
Reproducibility standard deviation, S_R, %	0.16	0.15	0.89	0.74	0.46	0.34	0.40
Reproducibility relative standard deviation, RSD_R, %	2.7	1.77	5.30	3.4	2.02	0.80	0.94
Reproducibility limit, R (=2.8S_R), %	0.44	0.41	2.50	2.08	1.30	0.97	1.11

calculated based on the results of the inter-laboratory study, excluding the statistical outliers were presented in Table 10 for total solids and Table 11 for total fat.

Table 10 showed average mean values for total solids ranging from 8.84% for light coconut milk to 99.25% for coconut milk powder. Values for S_r ranged from 0.02 to 0.49% and RSD_r ranged from 0.04 to 1.83%. Value for S_R ranged from 0.04 to 0.60% and value for RSD_R ranged from 0.21 to 2.24%. Values for r ranged from 0.05 to 1.38% and R ranged from 0.12 to 1.69%.

Table 11 showed average mean values for total fat ranging from 5.88% for light coconut milk to 42.90% for coconut milk powder. Values for S_r ranged from 0.03 to 0.76% and RSD_r ranged from 0.26 to 4.49%. Value for S_R ranged from 0.15 to 0.89% and value for RSD_R ranged from 0.80 to 5.30%. Values for r ranged from 0.10 to 2.12% and R ranged from 0.41 to 2.50%.

4. Conclusion

In global trading, dispute may arise if there is no agreed method for certain analytical item of certain commodity. For coconut milk and products, the standard limits for total solids and fat were established as detailed in the CODEX STAN 240-2003. However, no agreed methods are available. This study was conducted in order to address this problem. Various existing techniques were studied and compared. The resulting methods were the drying technique in hot air oven at $102 \pm 2\ ^{\circ}C$ for total solids and the solvent extraction method for fat. Both methods were validated in collaborative trials. The performance characteristics such as S_r, S_R, RSD_r, RSD_R, r and R were obtained. The methods were proposed to Codex Committee on Methods of Analysis and Sampling (CCMAS) and adopted as CODEX Type I method in 2009.

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